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TRANSLATION

DETERMINING ELECTRICAL PROPERTIES OF GRAPHITE AT HIGH TEMPERATURES

By

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FOREIGN TECHNOLOGY DIVISION

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FTD-TT- 62-1708/1+2 Date 14 Feb. 19 63

Determining Electrical Properties of Graphite at High Temperatures.

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L. F. Mal'tseva and E. N. Marmer

(All Union Scientific-Research Institute of Electrothermal Equipment)

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The data about the temperature dependence of specific electrical resistance of graphite, are very scant. And so, the Moscow Electrode Plant in prospects of its production speaks only about electrical resistance of graphite at room temperature whereby this value varies in broad intervals. For example, for graphite type "B" it constitutes 14-20 ohms.mm^2/m.

The purpose of this investigation - to determine the values of specific electrical resistance in dependence upon temperature (20-2500°) for industrial graphites, manufactured by the Moscow Electrode Plant.

Testing installation. Determination of temperature dependence of specific electric resistance of graphite was done on an installation, consisting of an electric furnace, vacuum system and electric part. A cross section of the working chamber is shown in fig.1.

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The installation consists of a water-colled body 1, upper 2 and lower 25 lids, also

having water cooling. In the central part of upper lid is situated a peeping window 5, through which it is possible to observe the sample 6 and to measure its temperature with a pyrometer. To protect the glass against overheating are set up screens 4, which are showed aside at the time of measuring.

In the lower furnace lids are placed bobbins (lugs) 21, 23 for the insertion of thermocouple 26 and potential tips 22, with which is measured the voltage drop on the sample. To supply current to the sample two copper water-cooled current conductors are used 14, situated on one axis one facing the other. A vacuum of the magnitude of 10^{-3} - 10^{-4} mm Hg is produced in the furnace by the forevacuum pump RVN-20 and diffusion pump TSVL-100, coupled with the installation by fastening device DU-85U.

Pressure in the system was measured with the aid of thermocouple tubes of LT-2 type and ionization tubes type LM-2. A VIT-1 type instrument served as vacuummeter.

Graphite samples were placed and secured in the vacuum chamber with upper lid removed by a special contrivance. In fig.1, is evident, how sample 6 200 mm long and 10 mm in diameter is secured.

With the aid of strip 9 and bolts 8, made of graphite, the samples are attached to a massive graphite plate 10. To this plate is also attached a copper plate 11, to which in turn is attached a strip of copper foil 17, connected with the current conductor. Graphite plates 10 were supported by stands 19 made of graphite, insulated from the body of the furnace by a ceramic plate 20. In order that the copper plates and the foil should not melt from direct irradiation (bombardment) of the sample, they were screened with molybdenum sheet 3 with a thickness of 0.2 mm.

In the zone of uniform temperature of the sample (it was determined by special experiments) were fastened potential clamps 7 for measuring voltage drop. The clamps represented shaped graphite strips, which with the aid of bolts and nuts made of graphite were pressed against the sample and to it were connected the potential ends made of tungsten wire 22.

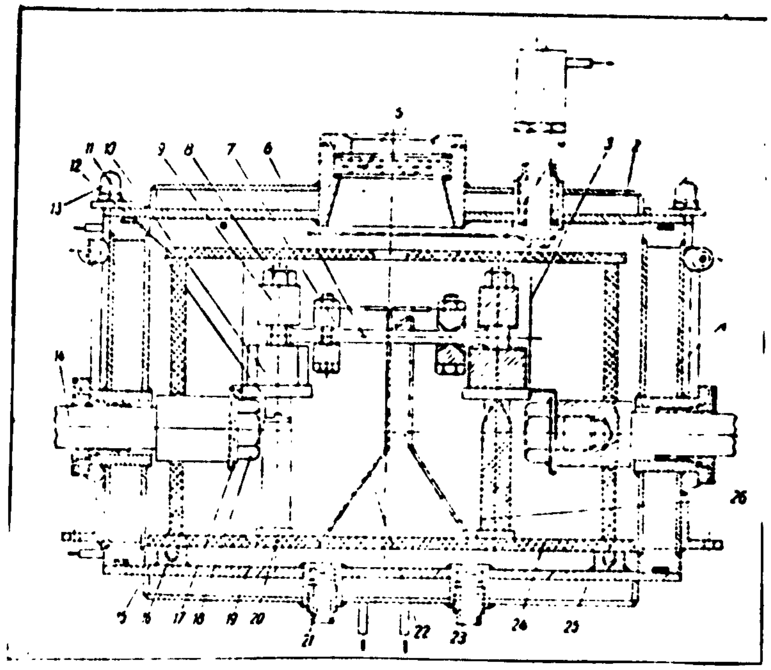

Fig.1. Installation for determining electric resistance of graphite at high temperature.

L*BODY OF FURNACE: 2*UPPER LID: 3-molybdenum shield; 4-shields for protecting the glass; 5-peeping window; 6-sample; 7- potential clamps (terminals); 8-graphite bolt; 9-strip; 10-graphite plate; 11-copper plate; 12-upper graphite shield; 13-releaseable bolts; 14-current conductors; 15-graphite shield; 16-steel ring; 17-copper foil; 18-steel bolt; 19-graphite stands; 20-ceramic plate; 21-lug for introduction of potential ends; 22-potential end; 23-lug for introducing thermocouple; 24-lower graphite shield; 25-lower lid; 26-thermocouple.

To reduce body and lid heating and to improve the conditions of measurin temperature in the interior of the vacuum chamber were mo nted graphite shields. The electric resistance of graphite samples was measured by the ammeter-voltmeter method. As is evident from fig.2. through the tested sample 4 was passed an electric current, which was measured with the aid of UTT-6 transformer and A_3 ammeter. The voltage drop on the sample 4 was measured with the aid of V_3 voltmeter. To regulate the voltage on the high side of the power transformer 3 was used a single phase autotransformer 2 type AOCK-25/0.5, enabling a smooth change in voltage in the range of from 0-20 v. The power step down transformer 3 tyoe OCY 40/0.5 offered the possibility of obtaining on the low side a voltage from 0 to 20 v at an amperage of 1400 amp.

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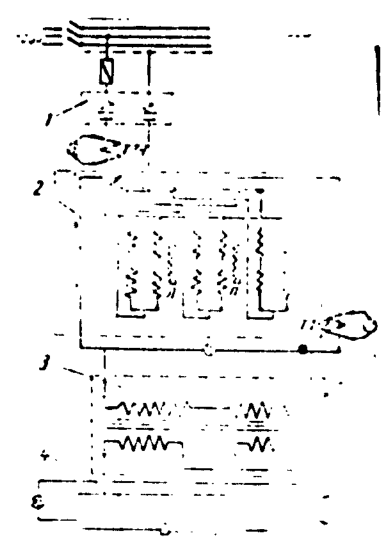

Fig.2.Electric circuit diagram of installation (designations see text)

The relative error of the experiment ,determinable by the type of selected scheme, class of accuracy of measuring instruments , class of accuracy of current transformers, has not exceeded 4%. Determination of the dependence of specific electric resistance of samples upon temperature , was done in a range of from 20-2500°.

To measure a temperature up to 1000° was used a chromel-alumel thermocouple. From 1000 to 2500° the temperature was measured with an optical micropyrometer of OPM-019 type with separate indicating instrument - milliammeter (class of accuracy of 0.2%). The relative error of measuring temperature by this instrument with consideration of the accuracy class of the milliammeter, absorption of parts of the rays by the peephole glass and correction for true temperature has not exeeded 2%.

The scale of dependence graphs of electric resistance of graphite upon the temperature was calculated according to the report[2] on the basis of analyzing experimentation errors.

Determining electric resistance. To conduct tests for the purpose of determining the dependence of specific electric resistance upon temperature were selected the most widely known types of graphite, manufactured by the Moscow Electrode Plant types A, B, C, and D.

Samples of type A graphite were cut out from billets in parallel and perpendicular directions relative to the axis of pressing. To remove the gas the sample was first heated to a temperature of 1800-2000°.

The time of exposure, necessary to equate the temperature on the working part of the sample, usually did not exceed 10 min. The temperature was measured through 100-150°. At each temperature value were measured several values of voltage and amperage, whereby one of these was measured at the end of the exposure period.

Specific electric resistance was calculated by formula :

$$\rho = R \cdot \frac{S}{l},$$

where $R = \frac{U}{I}$, and $\frac{S}{l} = \frac{\pi D^2}{4l}$ - value, constant for each sample, determined at the beginning of the experiment by the geometrical dimensions of the sample and the distance between the terminals.

The sequence of making the tests, exposure time and temperature ranges, in which the measurements were done, for samples of all types were selected approximately indentical. By the test results for each type of graphite were plotted dependence curves of specific electric resistance upon the mean temperature of the sample.

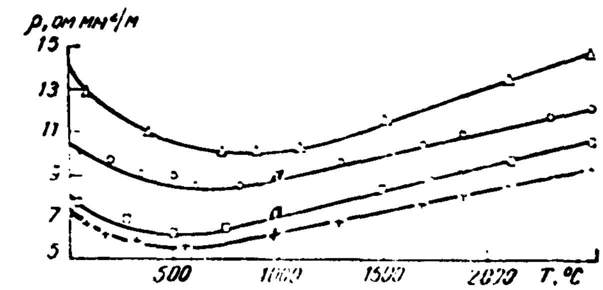

Fig.3. Dependence of specific electric resistance of type A, B, V graphite upon the temperature in vacuo 10^{-3} - 10^{-4} mm Hg: + - type A graphite with parallel arrangement of grains relative to the axis of deformation (mean value from 9 samples); □ - type A graphite with perpendicular arrangement of grains relative to the axis of deformation (mean value from 6 samples); o - B type graphite (mean value from 13 samples); Δ - Type C graphite (mean value from 8 samples).

Mean temperature was calculated by formula :

$$t_{mean} = \frac{t_p + t_{ts}}{2}$$

where t_p - surface temperature of the sample in °C, t_{ts} - temperature of the center in °C, and $t_{ts} = t_p + \frac{0.12 \cdot W_{spec} \cdot r}{\lambda}$, where W_{spec} - specific load per sample in watts

per square centimeter, r - radius of sample in cm, λ - coefficient of heat conduction of graphite at corresponding temperature (since for the type A graphite the coefficient of heat conduction at a temperature of 2500° equals 0.084 cal/cm.degrees.sec [1]

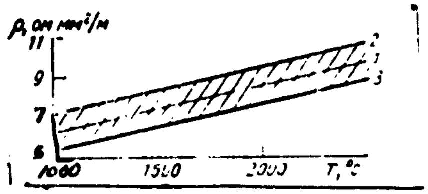

Fig.4. Dependence of specific electric resistance of A type graphite with parallel arrangement of grains relative to the axis of deformation upon temperature in vacuo 10^{-3} - 10^{-4} mm Hg;
1-mean value from 9 samples; 2-maximum values; 3-minimum values.

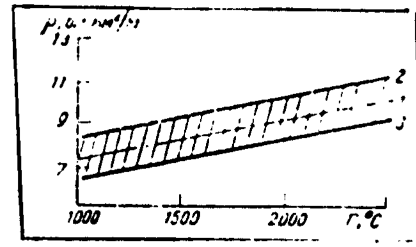

Fig.5. Dependence of specific electric resistance of type A graphite with perpendicular arrangement of grains relative to the axis of deformation upon the temperature in vacuo 10^{-3} - 10^{-4} mm Hg;
1-mean values from 6 samples; 2-maximum values; 3-minimum values;

In fig.3 are shown temperature dependence curves of electric resistance for A, B and C type graphites from 20 to 2500°. As is evident from graph fig.3. with a rise in temperature the electric resistance of graphite of all types decreases at first by approximately 30%, reaching its minimum value for A and B type graphites at a temperature of 400 - 600° and for C type at a temperature of 800-900°, and above a temperature of 1000° it rises linearly.

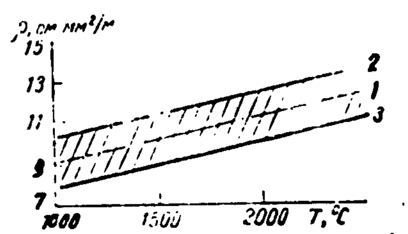

Fig.6. Dependence of specific electric resistance of B type graphite upon temperature in vacuo 10^{-3} - 10^{-4} mm Hg.
1-mean value from 13 samples; 2-maximum value; 3-minimum values.

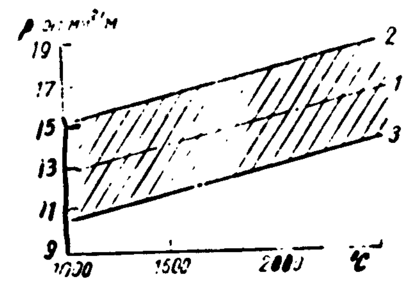

Fig.7. Dependence of specific electric resistance of B type graphite upon temperature in vacuo 10^{-3} - 10^{-4} mm Hg;
1-mean value from 8 samples; 2-maximum values ; 3-minimum values.

Consequently in the ranges of temperatures of 1000-2500° when calculating specific electric resistance it is possible to use formula:

$$\rho_t = \rho_{1000} + \alpha (t - 1000),$$

where ρ_{1000} - specific electric resistance at a temperature of 1000°.

In fig.4 are given temperature dependence curves of specific electric resistance of A type graphite (mean volumetric weight of samples 1.73 g/cm^3, porosity 21,7%) for samples, cut out in direction, parallel to the axis of pressing.

It is evident from this graph, that in the temperature range of 1000 - 2500° the specific electric resistance rises, changing its value from ρ_{1000} = 6.3 ohms . mm^2/m to ρ_{2500} = 9.35 ohms.mm^2/m, i.e. it rises approximately by 45%. Deviation of specific electric resistance values from the mean value in the investigated range of temperatures constitutes ± 15%.

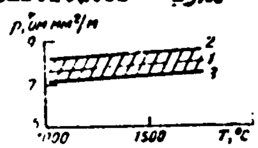

Fig.8.Dependence of specific electric resistance of D type graphite upon temperature in vacuo 10^{-3} - 10^{-4} mm Hg. 1-mean values from 2 samples; 2-maximum values; 3-minimum values.

In fig.5 is shown the temperature dependence of specific electric resistance of A type graphite for species, cut out in direction perpendicular to the axis of pressing (mean volumetric weight of species 1,78 g/cm^3, porosity 19,4%). It is evident from the graph that specific electric resistance upon temperature of 1000° to 2500° rises approximately by 40%. Deviation of absolute values of specific electric resistance upon mean value ± 15%.

For B type graphite (porosity 24%, mean volumetric weight of samples 1,67 g/cm^3) specific electric resistance from temperature of from 1000° to 2500° rises by approximately 40% (fig.6). Deviation of absolute valuesfrom mean values ± 15%.

In fig.7 is given the dependence of specific electruc resistance of C type graphite upon temperature (mean volumetric weight of samples 1,46 g/cm^3, porosity 33.6%). The specific electric resistance rises with temperature, it changes its mean value from ρ_{1000} = 12.9 ohms.mm^2/m to ρ_{2500} = 16.5 ohms.mm^2/m, i.e.it rises by approximately 35%. Deviation of values from mean value ± 20%.

In fig.8 is shown the temperature dependence of specific electric resistance

of D type graphite (mean volumetric weight of samples 1,43 g/cm^3, 35.5%). This type of graphite has a slight mechanical strength, and that is why the temperature dependence of specific electric resistance is presented at 1700°. Deviation of values from mean value 7%.

Conclusions

1. The greater disagreement in values of specific electric resistance in dependence upon temperature can be explained by the unstable technolo y of preparing the graphite and considerable heterogeneity of its structure.

2. Electro resistance of graphite of all types upon a rise in temperature from 20° drops at first by approximately 30%, reaching a minimum for types A, B and D in the temperature range of 400-600°, for type C - 800-900°. Above 1000° electric resistance rises linearly.

In the range of temperatures of 1000-2500° when calculating specific electric resistance it is possible to apply formula

$$\rho_t = \rho_{1000} + q(t - 1000)$$

For objects cut out in direction, parallel to axis of pressing, this formula has the form of :

a) for A type graphite: $q_t = (6.4 \pm 0.9) + 0.002\ (t-1000)$;

b) for B type graphite: $q_t = (9.2 \pm 1.4) + 0.002\ (t-1000)$;

c) for C type graphite: $q_t = (12.9 \pm 2.6) + 0.0024\ (t-1000)$;

d) for D type graphite in temperature range 1000-1700°

$q_t = (7.6 \pm 0.7) + 0.0009\ (t - 1000)$.

Absolute values of specific electric resistance for objects cut out in perpendicular direction to the pressing axis, is approximately by 20% higher.

3. Values of specific electric resistance of all types of graphite at a temperature of 20 and 1500° are approximately identical.

Literature

1. Technique of High Temperature (under editorship of Campbell) Foreign Lit., 1959.
2. M. Ye. Blanter, Method of Investigating Metals and Analysis of Experimentals Data, Metallurgizdat, Moscow, 1952.

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